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METHODS IN SOIL ANALYSIS

TECHNICAL BULLETIN

BY

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METHODS IN SOIL ANALYSIS

By FIRMAN E. BEAR and ROBERT M. SALTER.

INTRODUCTION.

Much importance has been attributed in recent years to the analysis of soils for total constituents. The literature covering the methods for such analysis, while rather extensive, is scattered, and in many cases the methods described are not well suited to rapid routine practice. In view of these facts it has been thought that the compilation and publication of the methods used and in part evolved in the soils laboratory of the West Virginia Agricultural Experiment Station may be of value to others desiring methods which are rapid and at the same time sufficiently accurate to meet the requirements of most soil investigations. The methods are presented in sufficient detail to permit their employment by those who have not had extensive experience in quantitative work.

In the development of these methods access has been had to the methods employed by the New York, Illinois, and Wisconsin experiment stations. Use has also been made of material from procedures as published by the Ohio, Tennessee, and other experiment stations. Much has been adapted from bulletin 422 of the United States Geological Survey.

Most of the methods herein described have been proved reliable by their successful use in several hundred analyses of various types of West Virginia soils.

CHOOSING SAMPLES.

In sampling any given area three composite samples are ordinarily chosen, representing different depths as follows: sample A, 0 to 6⅔ inches; sample B, 6⅔ to 20 inches; sample C, 20 to 40 inches. A one-inch soil auger is used in securing samples. Sample A is a composite of from 20 to 30 borings, so chosen as to furnish as nearly as possible a truly representative sample of the area in question. Samples B and C are composites of from 10 to 15 borings. As samples are taken in the field they are placed in clean cloth sacks and sent immediately to the laboratory where they are air dried and prepared for analysis.

PREPARATION OF SAMPLES FOR ANALYSIS.

The samples, after being air dried, are pulverized in a porcelain mortar to pass a 2-mm. sieve, care being taken not to pulverize any pieces of rock or shale. The material which fails to pass the sieve is weighed and its percentage of the whole sample calculated. This material is placed in a glass jar and labeled Discard, No....., A. B. or C.

The material which passes the sieve is thoroughly mixed and a sufficient amount selected by the method of quartering to fill an eight-ounce jar. This latter material is then further pulverized in an agate mortar so that all passes a 100-mesh sieve, no portion being rejected. All samples are stored in tight glass jars of eight- and sixteen-ounce sizes, with metal screw tops.

MOISTURE.

Five grams of 100-mesh soil are weighed into an aluminum dish provided with tight cover or into a low form weighing bottle with ground glass stopper. The soil is dried for five hours in an oven kept at 110° C. The dish or weighing bottle is cooled in desiccator, covered, and weighed.

DETERMINATION OF IRON, ALUMINIUM, TITANIUM, MANGANESE, CALCIUM, AND MAGNESIUM.

The procedure outlined for these determinations has been developed with the idea of eliminating the difficulties which arise when silica is determined in the same sample as the above-named elements. This has proved particularly advantageous at the West Virginia Experiment Station laboratory, since in many samples which have been analyzed for these elements the content of silica was not desired. The method depends on the decomposition of the sample by means of hydrofluoric acid.

Decomposition of Sample.

A one-gram sample of 100-mesh soil is treated in a platinum crucible of 35 cc. capacity with 5 cc. of hydrofluoric acid (48%) and ½ cc. of concentrated sulphuric acid. The mixture is slowly evaporated on a water or sand bath and the excess sulphuric acid driven off by inclining the crucible upon a triangle and cautiously heating the upper portion with a

low flame. When dry the crucible is ignited[1] until organic matter is burned off. From three to five cc. of hydrofluoric acid are added and again slowly evaporated on a water or sand bath. This is repeated until no gritty particles are noticed when residue is rubbed with a platinum spatula, two or three evaporations ordinarily being sufficient. Any fluorides are now changed to sulphates by treating with a few drops of H_2SO_4 and volatilizing over a free flame as previously described. The residue is treated with 5 cc. of concentrated HCl and transferred with water to a 500 cc. beaker. The volume at this point should be about 75 cc. The solution is boiled until residue dissolves.[2]

The acid present is partially neutralized with about 6 cc. of 1:1 NH_4OH (14% NH_3) and then 10% Na_2CO_3 solution is added drop by drop with stirring until the color of the solution tends to darken. The dropwise addition is continued from this point more slowly and with more thorough stirring between drops until the cloudiness produced by one drop seems to increase when stirred rather than to decrease. One or two drops of cencentrated HCl are now added to clear the solution.[3] Ten cc. of 30% sodium acetate solution are added and the volume made up to from 300 to 350 cc. with boiling water. The solution is brought to boiling and boiled from 2 to 3 minutes. After allowing the precipitate to settle it is filtered onto an 11-cm. ashless paper, and sucked dry[4]. It is not necessary to wash the precipitate at this point since it is transferred together with filter back to the original beaker and re-precipitated as follows: Five cc. of concentrated HNO_3 are added and the paper is broken up with a sharp glass rod. Water is added to make the volume up to 300 cc. The solution is brought to boiling and NH_4OH is added slowly until a slight excess is indicated by the odor of free NH_3. The solution is boiled one minute and allowed to stand until the

[1] Ignition burns off organic matter and also seems to help in the further decomposition of the silicates by hydrofluoric acid.

[2] A slight amount of insoluble residue is generally present. This probably consists, as found by Robinson (bulletin 122, United States Bureau of Soils), of barium sulphate, zircons and possibly other rare earths, and in some instances small amounts of undecomposed quartz. The amount of $CaSO_4$ derived from ordinary soils is such as to go entirely into solution when boiled with HCl. Any insoluble residue need not be filtered off since it does not interfere with the determination of total oxides, being weighed with oxides and again after pyro-sulphate fusion and reduction by H_2S with the crucible alone.

[3] If more than one or two drops of acid is required to clear the solution, it is better to add an excess of acid and repeat the neutralization with sodium carbonate.

[4] The basic acetate method is used for the first precipitation of total oxides, since there seems to be less tendency for manganese to be co-precipitated with this procedure than where the first precipitation is made with NH_4OH. It is open to possible objection that more aluminium passes into the filtrate than with the latter method. However, the subsequent procedure permits of the proper correction for aluminium.

precipitate settles. It is filtered onto an 11-cm. ashless paper and washed several times with hot 2% NH_4NO_3 solution (slightly alkaline with NH_4OH) with intermediate suction. The first and second filtrates are combined, made acid with HNO_3, and evaporated to dryness in a Non-sol or Jena beaker on the water bath. The ammonium salts are now volatilized by heating in an oven kept at 175° C., the beaker being kept covered with a watch glass[1]. The precipitate and paper are placed in a platinum crucible and the paper burned off at low heat. The precipitate is finally heated to constant weight over a Scimatco burner.

Determination of Iron, and Total Oxides.

The ignited oxides are brought into solution by fusing with 5 g. of potassium pyro-sulphate[2] and dissolving in hot water to which is added 1 cc. of concentrated H_2SO_4. The solution is transferred to a 250-cc. Erlenmeyer flask. The volume should be from 75 to 100 cc. The iron is reduced and dissolved platinum precipitated by passing in a slow stream of H_2S[3] and gradually raising the solution to boiling[4]. The passage of H_2S and boiling are continued until the precipitated sulphur flocculates when the flask is removed from the flame and allowed to cool somewhat, the gas flow being continued. The solution is filtered into a second Erlenmeyer flask and the precipitate washed with hot water. Precipitate and paper are transferred to the platinum crucible in which the fusion was made. This is then ignited and weighed[5]. Total oxides are secured by subtracting the weight obtained from that previously obtained for crucible plus total oxides. A cor-

[1] The removal of ammonium salts is necessary to facilitate complete recovery of aluminium by subsequent precipitation with manganese.

[2] During fusion with pyro-sulphate care is necessary to prevent loss by spattering. A convenient method is to place crucible (with lid) into a nichrome triangle held in a wooden handle. By holding this in one hand and rotating over flame the fusion may be controlled so as to prevent spattering. By means of a pair of tongs held in the other hand the lid may be removed frequently to note progress of fusion. Fusion at red heat for a few minutes is generally sufficient to produce a clear melt which indicates complete solution of oxides. By rotating the crucible so that the liquid melt congeals on the sides of the crucible, and then plunging it while still hot into cold water, the material is so loosened as to permit rapid solution in hot water.

[3] Passage of H_2S into solution is accomplished by fitting Erlenmeyer flask with 2-hole rubber stopper, one hole carrying a glass tube, the lower end of which is drawn to a fine point and extends to the bottom of the flask. This serves as the inlet tube and is connected to Kipp generator. The other hole of stopper carries a short L-tube, one arm of which extends just below the stopper. This facilitates testing for complete removal of H_2S with lead acetate paper after reduction.

[4] When work can be so arranged, saturation of solution in the cold with H_2S, stoppering the flask, and allowing it to stand over night results in complete reduction and in the formation of a precipitate which is easily filtered.

[5] The ignited residue consists of platinum dissolved during pyro-sulphate fusion, together with materials which escaped decomposition by hydrofluoric and sulphuric acids.

rection is obtained later for the amount of aluminium in the original filtrates. H_2S is again conducted through the hot filtrate obtained as previously indicated to reduce any iron which may have been oxidized by contact with air during filtration[1]. The flask is then disconnected and CO_2[2] passed through the boiling solution until the H_2S is entirely expelled. This point can be determined by testing with lead acetate paper[3]. The flask is cooled by placing in cold water, continuing the passage of CO_2. When cool the iron is immediately titrated with N/25 $KMnO_4$ solution and calculated as grams of Fe_2O_3.

Determination of Titanium.

The solution in which iron was determined is transferred to a 250-cc. graduated flask, 10 cc. of concentrated H_2SO_4 are added and the solution is cooled to room temperature. Five cc. of 3% H_2O_2[4] (free from fluorides) are added, the solution is brought to mark with water and thoroughly mixed. The amount of titanium is determined by comparing the color produced with that of a standard $Ti_2(SO_4)_3$ solution similarly peroxidized, using a Dubosc colorimeter[5]. The titanium is calculated as grams of TiO_2.

Determination of Manganese.

The residue obtained by the evaporation of the filtrates from the total oxides is dissolved in about 75 cc. of water. Twenty cc. of a saturated solution of bromine and sufficient NH_4OH to dispel the bromine color are added and the solution is brought to boiling. In order to insure complete precipitation the solution is cooled, more bromine water and NH_4OH are added, and it is again brought to boiling. If necessary this operation is repeated. The flocculant precipitate is filtered upon an ashless paper and washed with hot

[1] A slight opalescence due to sulphur may appear at this point but does not interfere with the subsequent permanganate titration.

[2] Generated in Kipp from marble. Gas is passed through wash bottle containing acid $CuSO_4$ solution to free from H_2S.

[3] Prepared by soaking filter paper in 3% lead acetate solution and drying.

[4] Hydrogen peroxide should be free from fluorides which bleach the color produced. Sulphuric acid tends to counteract the bleaching effect of alkali sulphates present (see Merwin, Am. Jour. Sci., Vol. 28, p. 119).

[5] The standard titanium solution is conveniently made up by dissolving sufficient $Ti_2(SO_4)_3$ in dilute H_2SO_4 so that the resulting solution contains titanium equivalent to 0.0100 grams TiO_2 per 10 cc. The exact strength of this solution is determined by precipitation of known volume with NH_4OH, igniting and weighing up the TiO_2 formed. The color standard is made up by oxidizing 10 cc. of above-mentioned solution with H_2O_2 and diluting to 250 cc.

water[1]. Paper and precipitate are transferred to a previously weighed platinum crucible, ignited, and weighed. The weight of precipitate represents Mn_3O_4 plus the Al_2O_3 recovered from the original filtrates. The precipitate is brought into solution by fusion with about 1 gram of potassium pyrosulphate and dissolving in from 50 to 75 cc. of hot water. The solution is transferred to a 250-cc. graduated flask and made strongly acid with H_2SO_4. Ten cc. of .2% $AgNO_3$ solution are added for each milligram of metallic Mn, and this followed by about 1 gram solid ammonium persulphate. The flask is placed on a steam bath until a pink color appears when it is removed and placed in cold water. When color is fully developed the flask is filled to the mark and contents are thoroughly mixed. The amount of manganese is determined by comparison in Dubosc colorimeter of the color obtained with that of a standard[2] prepared by acidifying a standard $KMnO_4$ solution, reducing with sulphurous acid, re-oxidizing as with the regular sample, and diluting appropriately. The amount of Mn determined is calculated as Mn_3O_4 and this subtracted from the weight of $Mn_3O_4 + Al_2O_3$ previously obtained. The difference represents the Al_2O_3 recovered from the filtrate from the total oxide precipitation and is added to the total oxides previously determined.

Determination of Aluminium.

Aluminium is determined as Al_2O_3 by subtracting the sum of the weights of Fe_2O_3, TiO_2, and P_2O_5 from the corrected weight of total oxides (P_2O_5 determined in separate sample).

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[1] A different procedure, permitting the determination of manganese gravimetrically, may be used instead of the colorimetric method outlined. However, it is more difficult to get accurate results gravimetrically due to difficulty in completely separating the Al and Mn. The procedure from this point is as follows: Paper and precipitate are transferred to a small beaker, paper is broken up with glass rod, and precipitate is dissolved in sulphurous acid. The solution is heated to boiling and enough NH_4OH added to make just alkaline to litmus. It is filtered immediately and the precipitate washed several times with hot 2% NH_4NO_3 solution. Precipitate and paper are transferred to weighed platinum crucible and ignited. Weight of residue found is added to that of total oxides previously determined. The manganese is determined in the filtrate by precipitation with NH_4OH and bromine as before. (The addition of sufficient strong KOH solution to keep any traces of $Al(OH)_3$ in solution tends to give better results). The flocculent precipitate, which should be almost black, is filtered on ashless paper, washed thoroughly with hot water, transferred with paper to weighed platinum crucible and ignited to constant weight. The residue consists of Mn_3O_4 and is calculated to MnO. Factor = 0.93006 Log = $\bar{1}$.06851.

[2] The standard is conveniently made to contain .0025 g. MnO per 250 cc.

Determination of Calcium.

The filtrate from the manganese precipitation is regulated to a volume of 150 cc. and heated to boiling. Ten cc. of saturated ammonium oxalate are added slowly, the solution being kept boiling meanwhile. The solution is further boiled for 15 minutes and filtered through a close-textured filter (C. S. & S. Blue Ribbon), the precipitate being washed free from oxalates with hot water. The precipitate is dissolved into an Erlenmeyer flask by passing warm dilute H_2SO_4 (1:10 at about 70° C.) through the filter. The oxalic acid formed is titrated warm with N/25 $KMnO_4$ and the calcium calculated as CaO. One cc. N/25 $KMnO_4 = 0.00112$ g. CaO. The precipitation of calcium is never complete, as a certain amount of calcium oxalate is always required to saturate the solution in which the precipitation is made. This amount is practically constant and equal under the aforementioned conditions to 0.0007 g. CaO.

Determination of Magnesium.

To the cold filtrate from the Ca precipitation are added 5 cc. of 10% solution of microcosmic salt ($NaNH_4HPO_4$) and 10 cc. of NH_4OH (1:1). Precipitation is started by stirring and rubbing the walls of beaker with a glass rod. After being allowed to stand over night the precipitate is brought upon a close-textured filter paper, washed with cold 2% NH_4OH, and dissolved back into the original beaker with warm, dilute HCl (1:10) and hot water[1]. The magnesium is re-precipitated in a volume of about 75 cc. as follows: The solution is made neutral or just alkaline to litmus with NH_4OH and 1 cc. of 10% $NaNH_4HPO_4$ solution is added. After standing 15 minutes, 10 cc. of concentrated NH_4OH (28% NH_3) are added and the solution is allowed to stand two hours. The precipitate is filtered upon a close-textured ashless paper and washed with 2% NH_4OH solution. Filter and precipitate are transferred to weighed platinum crucible, ignited, and weighed. The calcium which failed to precipitate as oxalate appears in the ignited residue as $Ca_3(PO_4)_2$ and necessitates a correction of .0013 g. equivalent to the .0007 g.

[1] A re-precipitation is necessary since if the first precipitate is ignited directly the residue may not contain all the magnesium as pyro-phosphate (See Hillebrand, bulletin 422, United States Geological Survey, p. 123).

CaO added to the calcium obtained by titration[1]. The magnesium exists in the residue as $Mg_2P_2O_7$ and is calculated as MgO. Factor = 0.36207, Log. = $\bar{1}$.55879.

Blank Determination.

A blank is run using the same amounts of reagents and the appropriate corrections are applied[2].

DETERMINATION OF POTASSIUM.

The fusion for potassium is made according to the method of Laurence Smith for total alkalies. Potassium is precipitated as chloroplatinate without previous removal of calcium which is subsequently removed by means of acidulated alcohol wash. (See Moore, J. Am. Chem. Soc., Vol. XX, pp. 340-343.) Potassium is weighed as chloroplatinate.

Solutions.

Platinic Chloride Solution.—Eighteen grams of the salt are dissolved in water, made slightly acid with HCl, and diluted to one liter.

Acidulated Alcohol Wash.—To 3000 cc. of 95% alcohol are added 230 cc. of HCl, sp. gr. 1.20. HCl gas is conducted into the mixture until it shows a normality of approximately 2.25 by titration. The mixture must be kept cool during this process. The HCl gas is generated by treating c. p. NaCl with concentrated sulphuric acid.

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[1] The amount of CaO precipitated with magnesium was found to be quite uniform in amount and equal under outlined conditions to .0007 g. CaO. This correction is the same as that found necessary by Robinson (bulletin 122, United States Bureau of Soils). The calcium occurs in the magnesium residue as $Ca_3(PO_4)_2$ (See Hillebrand, bulletin 422, United States Geological Survey, p. 127).

The exact amount of this correction for any given set of conditions may be determined by method outlined by Hillebrand as follows:

The magnesium pyro-phosphate is dissolved in a little dilute sulphuric acid, and enough absolute alcohol added to make from 90 to 95% of final volume. After several hours the fine precipitate of calcium sulphate is brought on a close-textured filter and washed free of phosphoric acid with 95% alcohol. It is then dissolved in hot water, acidified with hydrochloric acid, made alkaline with ammonia, heated to boiling and the calcium precipitated by adding a few crystals of ammonium oxalate. In a short time it may be filtered, ignited, and weighed as CaO.

[2] A blank run on the reagents used in this laboratory gave the following values:

Fe_2O_3	=	.0007 gram.
Al_2O_3	=	.0011 gram.
TiO_2	=	none.
MnO	=	none.
CaO	=	.0004 gram.
MgO	=	.0002 gram.

80% Alcohol Wash.—Made by diluting 95% alcohol to a specific gravity of .864 at 15.6° C.

Gladding Wash.—Made by dissolving 200 grams of ammonium chloride in water and diluting to 1 liter.

Procedure.

Five-tenths of a gram of 100-mesh soil is weighed and thoroughly ground with .5 gram of NH_4Cl in agate mortar. Four grams of calcium carbonate are added and the mixture is ground until well mixed. Sufficient calcium carbonate is placed in a 35-cc. platinum crucible to cover the bottom and the mixture added by first brushing onto a sheet of glazed paper and then transferring to crucible. About .5 gram of calcium carbonate is ground in the mortar, brushed onto the paper and transferred to crucible together with brushings from paper on which mortar has stood. After tapping to settle contents, the crucible is covered with lid and placed in a hole in an asbestos board held in an inclined position. The hole in the asbestos board should be of such size as to allow about two-thirds of the depth of the crucible to project below the board. The crucible is first heated with a low flame until the odor of ammonia is no longer noticeable (10 to 15 minutes) when the flame is turned up and the crucible heated at the full heat of a Scimatco burner for 45 minutes. The lid of the crucible should stay well below red heat during fusion. After cooling, the fused mass is loosened with a glass rod and transferred to a porcelain casserole. The lid is washed with hot water and the crucible filled with hot water and allowed to stand for a few minutes. The contents of the crucible are washed into the casserole and the fusion is crushed with an agate pestle. The casserole is covered with a watch glass and digested on the water bath for two hours or allowed to stand over night. After thorough slaking, the mixture is stirred and the liquid decanted into a coarse alundum filtering crucible[1] receiving the filtrate in a 400-cc. beaker. Boiling water is added, the mixture stirred and again decanted into filter. This is repeated two or three times when the whole fusion is transferred to the crucible and washed with hot water until the filtrate measures about 300 cc. Ten cc. of HCl, sp. gr.

[1] Filtering through a coarse alundum filtering crucible was found preferable to filtering through paper since the former eliminates a slimy material which seems to be derived from the action of caustic lime upon the filter paper and which is difficult to wash out in the final filtration.

1.20, are added and the filtrate is evaporated to dryness on the water bath, taken up with hot water and filtered into 150-cc. beaker. The filter is washed until filtrate nearly fills beaker. One cc. of concentrated HCl and 5 cc. of platinic chloride solution are added and the filtrate is evaporated on the water bath until residue just solidifies. The residue is treated with from 15 to 20 cc. of acidulated alcohol and stirred until the calcium chloride all dissolves. The liquid is decanted into previously weighed Gooch crucible, using disc of "S. & S. Blue Ribbon" filter paper instead of asbestos mat as filtering medium[1]. The precipitate is transferred to crucible and thoroughly washed with 80% alcohol. It is then washed 10 or 12 times with small amounts of Gladding wash, followed by 10 or 12 washings with 80% alcohol. The crucible is placed in drying oven at 115° C. for 20 minutes, then cooled and weighed. A blank is run on the reagents used and subtracted from the weight of K_2PtCl_6 obtained.

$$\text{Factor } \frac{K_2}{K_2PtCl_6} = 0.1608 \quad \text{Log.} = \bar{1}.20643.$$

DETERMINATION OF PHOSPHORUS.

Solutions.

Magnesium Nitrate Solution.—1000 grams of magnesium nitrate are dissolved in 1000 cc. of water.

Aqua Regia.—Three volumes HCl, sp. gr. 1.19, and one volume HNO_3, sp. gr. 1.42 are mixed and allowed to stand until fumes cease to be given off.

Molybdate Solution.—100 grams of molybdic acid, Baker's c. p. 99% are dissolved in 144 cc. of ammonium hydroxide, specific gravity 0.90, and 271 cc. of water. The solution thus obtained is poured slowly with stirring into 489 cc. of nitric acid, specific gravity 1.42, and 1148 cc. of water. A few drops of dilute sodium phosphate solution are added and the molybdate solution allowed to stand several days in a warm place. The clear solution is then siphoned off and preserved in a glass stoppered bottle.

[1] Discs of "S. & S. Blue Ribbon" filter paper are used in preference to asbestos since they permit more rapid filtration, are less liable to lose in weight, require less washing, and dry more quickly. After drying, the crucible must be handled carefully to prevent loss of precipitate.

Wash Solutions.—No. 1 solution containing approximately 1% nitric acid and 3% ammonium nitrate is made by adding sufficient water to 30 grams of ammonium nitrate and 10 cc. of nitric acid, sp. gr. 1.42, to make 1 liter of solution. No. 2 solution containing 3% ammonium nitrate is made by dissolving 30 grams ammonium nitrate in one liter of water.

Standard HNO_3 Solution.—Solution is made to contain 0.009339 gram HNO_3 per cc. equivalent to 0.0002 gram phosphorus as ammonium-phospho-molybdate.

Standard NaOH Solution (Free from carbonates).—Solution is mde to contain .005929 gram NaOH per cc., equivalent to 0.0002 gram phosphorus as ammonium-phospho-molybdate. Enough $BaCl_2$ is added to precipitate any carbonates as $BaCO_3$.

Phenolphthalein Solution.—A 5% phenolphthalein solution in 95% alcohol.

Procedure.

Five grams of 100-mesh soil are weighed and transferred to a 100-cc. porcelain dish. To this 5 cc. of $Mg(NO_3)_2$ solution are added, the mixture brought to dryness on a water bath, and dried one hour in electric oven at from 110° to 120° C. It is then ignited until all organic matter is burned off, the ignition being started with a low flame and finished at low red heat. When cool, the mass is moistened with a small amount of water and broken up with a glass rod. The dish is covered with a watch glass and 20 cc. of aqua regia are added through the lip after which the mixture is digested on boiling water bath for two hours with occasional stirring to break up lumps. The material is transferred to a 250-cc. graduated flask by washing through a glass funnel, and, after cooling to room temperature, made up to mark with water. After thorough shaking the solution is either filtered through a dry folded filter into a dry beaker or allowed to stand over night. If filtered, 200 cc. of the clear filtrate, or in the latter case, 200 cc. of clear supernatant liquid are pipetted off and evaporated to dryness on water bath in beaker of 400 cc. capacity. The residue is taken up with 10 cc. of dilute nitric acid (1:4)and again brought to dryness, after which it is dehydrated for 1 hour in oven at from 110° to 120° C. The residue is taken up with 5 cc. HNO_3, sp. gr. 1.42, and hot water, and filtered with suction into 250-cc. Erlenmeyer flask, thoroughly washing filter with hot water. The filtrate is evaporated to a volume of approximately 50 cc. on water

bath[1]. Five grams of ammonium nitrate are added and when in solution 20 cc. of molybdate solution are added. The flask is shaken on a mechanical shaker for 15 minutes and then allowed to stand in oven at from 60° to 65° C. for two hours. The precipitate is filtered upon a 9-cm. filter (S. & S. Blue Ribbon filter or paper of similar quality) and washed three times with wash solution No. 1. The precipitate is dissolved back into original flask with warm ammonium hydroxide solution (1:9) and the filter washed thoroughly with hot water[2]. If filtrate exceeds 50 cc. it is reduced to this volume on the water bath. Three grams of ammonium nitrate are then added, followed by 3 cc. of molybdate solution. (With

[1] The greatest difficulty in the determination of phosphorus lies in the proper adjustment of concentrations in the precipitating solution so as to bring about complete precipitation of all phosphorus and yet produce a precipitate free from molybdic acid, and of good physical condition. Complete precipitation is favored by high temperature, large excess of molybdate solution, presence of NH_4NO_3, and long standing. (Hibbard, J. Ind. & Eng. Chem. Vol. 5, pp. 998-1010.) However, these factors also all tend to cause the precipitation of molybdic acid. To counteract this tendency for MoO_3 to separate, a certain amount of HNO_3 is used, this acting through its solvent effect on MoO_3. Too great an excess of HNO_3, however, causes solubility of ammonium-phospho-molybdate and low results.

In working with soils there are present in some cases large amounts of iron salts which, as shown by Hibbard, tend to cause incomplete precipitations, but this may be remedied by a large excess of molybdate solution. Robinson (See J. Ind. & Eng. Chem. Vol. 8, pp. 148-151) states that soils contain appreciable amounts of vanadium (0.01 to 0.08%) which cause low results when precipitation is carried out under the conditions necessary for the determination of phosphorus by direct titration of the yellow precipitate. This he shows to be due to incomplete precipitation of phosphorus and remedies by reduction of vanadium with ferrous sulphate and precipitation of phosphorus in the cold with mechanical agitation. However, he writes. "By taking precautions to make the precipitation of phospho-molybdate complete by means of comparatively large excess of reagents, and by digestion and mechanical agitation, the influence of the amount of vanadium ordinarily found in soil can be avoided without reducing the vanadium, provided the yellow precipitate is converted to magnesium ammonium phosphate." The error due to presence of vanadium is probably much decreased if not entirely eliminated by a second precipitation if the conditions of the first precipitation are such as to induce complete precipitation of phosphorus.

On account of the difficulty in overcoming the above-mentioned errors, and at the same time obtaining a precipitate free from MoO_3 and containing all the phosphorus as ammonium-phospho-molybdate it was thought advisable to make two precipitations, the first being made under conditions inducing complete precipitation but not tending toward absolute purity of precipitate, while the second precipitation, made in the absence of all but traces of impurities, would be more easily adjusted to obtain complete precipitation with freedom from MoO_3.

Conditions of first precipitation:

Volume — 70 cc.
Phosphorus — .0008 to .0080 gram.
$(NH_4)_2MoO_4$ — approximately 2%.
HNO_3 — approximately 8%.
Mechanical shaking, 15 minutes in cold.
Digestion at 60° to 65° C. for two hours.

Conditions of second precipitation:

Volume — 50 cc.
Phosphorus — .0008 to .0080 gram.
$(NH_4)_2MoO_4$ — 0.4 to 1% (About double theoretical amount).
HNO_3 — 2%.
NH_4NO_3 — 6%.
Precipitation at exactly 60° C. with mechanical shaking for 3 minutes.

EQUATION:

$$NH_4H_2PO_4 + 12NH_4MoO_4 + 22HNO_3 = (NH_4)_3PO_4(MoO_3)_{12} + 22NH_4NO_3 + 12H_2O$$

[2] The appearance of a feathery white precipitate at this point indicates incomplete removal of bases, iron, aluminium, and more particularly titanium, which separate, probably as phosphates, in the alkaline solution. Robinson (previous reference) found that a second precipitation reduced the amount of feathery precipitate to a practically negligible quantity.

soils high in phosphorus, as indicated by a large amount of yellow precipitate in first precipitation, from 5 to 7 cc. of molybdate solution should be used). If a precipitate appears at this point it is re-dissolved by the addition of a little ammonium hydroxide. The solution is then brought to 60° C. (conveniently kept in oven at that temperature) and concentrated nitric acid added from burette, a drop at a time, until the color of solution turns permanently yellow, after which just one cc. more is added and the flask shaken for three minutes. The solution is filtered immediately and the precipitate washed three times with wash solution No. 1, three times with wash solution No. 2, and finally three times with water. The precipitate and filter are returned to flask in which precipitation was made and 25 cc. of standard NaOH solution are added from an automatic overflow pipette, rotating the flask so as to dissolve any precipitate adhering to the inner surface. (If soil contains more than 0.100% phosphorus, 50 cc. of NaOH should be added.) The sides of flask are washed down with distilled water and the filter is broken up with a glass rod. When yellow precipitate is all dissolved, 40 cc. of CO_2-free distilled water are added and the solution is titrated with standard HNO_3 solution using phenolphthalein indicator. When approaching the end point in the titration the flask is stoppered with rubber stopper and shaken to break up the filter thoroughly after which the titration is completed[1].

$$\text{Percent } P = \frac{\text{cc. of NaOH} - \text{cc. of } HNO_3{}^{2}}{200}$$

DETERMINATION OF TOTAL NITROGEN.

Solutions.

Alkali Solution.—Ten pounds Greenbank's alkali and 125 grams K_2S are dissolved in 5 liters of water.

Standard Acid.—5/14 normal H_2SO_4 is used. Arranged to deliver 10 cc. from automatic overflow pipette.

Standard Alkali.—N/14 NaOH is used, 1 cc. equivalent to 1 mg. N.

Procedure.

Ten grams of 100-mesh soil[3] are digested in 800 cc. Kjeldahl flask with 20 cc.[4] concentrated H_2SO_4 and 0.4 g.

[1] A blank determination should be run, using the same quantity of reagents as in regular determination.

[2] Equation used in calculation:

$$(NH_4)_3PO_4(MoO_3)_{12} + 23NaOH = (NH_4)_2HPO_4 + 11H_2O + NH_4NaMoO_4$$

[3] The use of soil ground to 100 mesh largely eliminates bumping during digestion.

[4] Soils high in clay or organic matter may require 25 cc. H_2SO_4 with subsequent use of 60 cc. of strong alkali.

metallic mercury (6 drops from burette) at low heat for 20 minutes. Five to ten grams K_2SO_4 are added and the digestion continued at full heat for 1½ hours or until residue is white. When cool 300 cc. of water and a few pieces of mossy zinc are added, 50 cc. of alkali solution poured down neck of flask, and the ammonia distilled into 10 cc. of 5/14 normal H_2SO_4. The distillate, which should measure about 200 cc., is titrated with N/14 NaOH, using alizarin red indicator. A blank is run on the reagents. Duplicates should check within 0.1 cc. equivalent to 0.001%.

$$\text{Percent N} = \frac{\text{Titration of blank} - \text{Titration of sample.}}{100}$$

DETERMINATION OF TOTAL CARBON.

The method used[1] is an adaptation with modifications of the method described by Fleming[2] for the rapid determination of carbon in iron and steel. It depends upon the direct combustion of the soil in a current of oxygen, the gases being dried by phosphoric anhydride and the carbon dioxide absorbed in soda-lime and determined by weight.

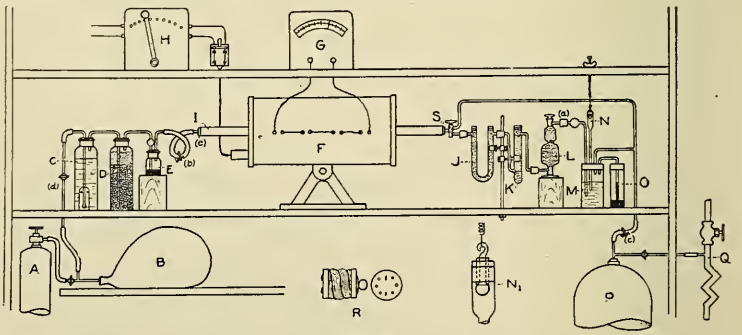

FIG. 1.

A—High Pressure Oxygen Tank, 150 gal., 1800 lbs. pressure. (S. S. White Dental Co., c. p. oxygen).
B—Rubber Bag, 1 gal. capacity. C—30 percent KOH Solution.
D—Granular, Anhydrous $CaCl_2$ (below), Separated by Layer of Asbestos Fiber from 20-mesh Soda-Lime (above).
E—Mercury Valve Bottle. F—Electric Combustion Furnace. H—Rheostat.
I—Silica Combustion Tube, Glazed, 24 in. long by ⅞ in. inside diameter.
J—U-tube Containing 2-mm. Granulated Zinc.
K—P_2O_5 Supported on Glass Wool. L—Fleming Soda-Lime Absorption Bulb.
M—Suction Bottle. N—Safety Valve, Ground Glass (shown below at N_1).
O—Mercury Suction Gauge. P—10 gal. Suction Tank.
Q—Water Suction Pump. R—Asbestos Plug. S—Two-way Stopcock.

[1] Published in Jour. Ind. & Eng. Chem., July, 1916, Vol. 8, pp. 637-639.

[2] The Iron Age, Vol. 93, pp. 64-66.

In the apparatus shown in Fig. 1, the gas enters bottle C, through a Folin absorption tube. The KOH solution frees the oxygen from any traces of carbon dioxide. D dries the gas and insures freedom from carbon dioxide. The mercury valve bottle prevents gas from backing into bottles C and D. The furnace F is an Eimer and Amend, having replaceable heating units, but is modified by installing 4 platinum-nichrome thermocouples connected in series to galvanometer G, so as to show the furnace temperature at all times; the scale on the galvonometer is calibrated by comparison with a standard pyrometer. The tube I contains, just within the exit end of the furnace, 5 in. of coarsely granular cupric oxide, held in position by 2 plugs of asbestos fiber. J, containing granular zinc, serves to stop sulphur, chlorine or acid fumes; it also acts as a filter. K removes moisture from the gases, and as the P_2O_5 liquefies it is absorbed by the glass wool, more anhydride being added from above. The lower portion of the absorption bulb L is filled with alternate layers of Baker's 20- and 40-mesh soda-lime, the quantity used being sufficient for 60 or more determinations of total carbon in average soils. The upper portion contains P_2O_5, which insures the gases leaving the bulb with the same moisture content as on entering. The valve N prevents accidental drawing of mercury from O into M and facilitates regulation of suction. The suction tank P gives more uniform suction than the pump alone, and one exhaustion serves for 15 or more determinations. R is made by joining two perforated discs of asbestos board by means of a stiff nichrome wire (B. & S. gauge 20) so as to leave a loop at one end by which the plug may be withdrawn from the combustion tube. Between the two asbestos discs is some loose asbestos fiber, held in place by a spiral of nichrome wire. The stop-cock S permits the drawing of oxygen through the combustion tube without allowing it to pass through the absorption end of the train. The whole apparatus is permanently set up in an electrically lighted case with sliding glass doors.

Procedure.

A two-gram sample of soil[1] is weighed, mixed with 2 to 3 g. of 40-mesh alundum and transferred to an alundum boat[2]. Before introducing the sample into the combustion

[1] With soils containing over 5 percent total carbon, a one-gram sample is sufficient.

[2] Convenient size of the boat for two-gram sample is 3¾ in. by ⅝ in., outside dimensions. Alundum mixed with soil increases porosity and insures access of oxygen to all parts. "R. R. Alundum, alkali-free, especially prepared for carbon determinations," is used.

tube[1] it is necessary to see that the exit tube of the absorption bulb is disconnected from the suction bottle at the point (a), that the two-way stopcock S stands in a position to permit gas entering the absorption end of the train, and that the screw clamps (b) and (c) and stopcock (d) are closed. The furnace[2] should stand at a temperature of from 925° to 950° C. The boat is introduced into the end of the combustion tube, followed by asbestos plug R. Both are then pushed to the center of the furnace by means of a stiff nichrome wire and connection (e) is quickly made. After a few seconds to allow the gas immediately produced to escape, screw clamp (b) is opened and connection (a) closed. Sufficient suction, measured by mercury gauge O, to produce somewhat more than desired rate of gas flow (previously determined) is then applied. The gas flow is regulated by adjusting stopcock (d) so that 750 cc. to 1000 cc. of oxygen pass through the apparatus in 20 minutes, which is sufficient time to complete combustion[3]. The temperature is maintained at from 925° to 950° C. throughout the combustion. The absorption tube[4] is finally disconnected, both inlet and exit closed, allowed to stand on the balance pan 15 minutes and weighed[5].

Duplicate determinations should check within .01% total carbon, equivalent to about 0.0007 g. CO_2 on a two-gram sample.

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[1] Before starting a single determination, or a series of determinations, the apparatus should be connected up and sufficient oxygen passed through to burn out any carbon contained in the asbestos plugs and to sweep out the air originally in the train. The further passage of oxygen should produce no increase in the weight of absorption bulb.

[2] Care must be observed to prevent the furnace attaining a temperature of much over 1000° C., as cupric oxide fuses at 1064° C. and when this occurs the silica tube slags with the fused oxide and invariably cracks on cooling. A temperature of over 825° C. is necessary to insure decomposition of carbonates.

[3] Drawing the gas through by suction is found preferable to forcing it through by pressure, it being much easier to prevent leakage. Any error from this source would be comparatively insignificant as the leakage would be inward rather than outward. The content of CO_2 in ordinary air is such that it would require an inward leakage of approximately 160 cc. to cause an increase of 0.0001 g. in the weight of the absorption bulb. All connections, where possible, were made of rubber stoppers, these being less liable to develop leaks than connections of rubber tubing.

[4] The Fleming absorption bulb was found to be very efficient, it being possible to increase the rate of gas flow 5 to 8 times that actually used without danger of incomplete absorption of CO_2.

[5] Due to the rather large size of the absorption bulb, some precaution is necessary in weighing to prevent errors due to differences in the amounts of moisture condensed on the surface of the bulb and to changes in temperature and atmospheric pressure. By using a second bulb or glass bottle of approximately the same weight, containing about the same quantity of soda-lime, as a tare in weighing, these errors are rendered insignificant.

DETERMINATION OF TOTAL SULPHUR.

Procedure.

Two grams of 100-mesh soil are mixed in a platinum crucible with 7 grams of anhydrous Na_2CO_3 of low sulphur content and .5 gram KNO_3. The crucible is covered with lid and placed in a hole in an asbestos board supported in a horizontal position. The hole in the asbestos board should be just large enough to permit about three-fourths of the crucible to extend below the board. The board itself should be large enough to prevent, as far as possible, the gases of the flame from coming in contact with the top of the crucible. The mixture is heated to quiet fusion with the full heat of a Scimatco burner. The crucible is now grasped in the tongs and rotated in such a way as to cause the melt to solidify upon the sides of the crucible, and while still hot, plunged into cold water to loosen the melt. Crucible and lid are placed in a 400-cc. beaker, covered with boiling water, and allowed to stand with occasional stirring until melt dissolves. The solution is then filtered onto an 11-cm. filter into a 400-cc. beaker and the residue washed with water until the filtrate measures 250 to 300 cc. A drop of alcohol is added to the cold filtrate to reduce any maganese, and then HCl from a burette with stirring until the solution is acid to litmus, avoiding an excess. The solution is heated to boiling and 5 cc. of 10% $BaCl_2$ solution added with stirring. Boiling is continued for 5 minutes and the solution allowed to stand over night. The precipitated $BaSO_4$ is filtered onto a close-textured ashless paper (S. & S. Blue Ribbon) and washed with hot water until free from chlorides. Paper and precipitate are transferred to weighed platinum crucible and ignited and sulphur weighed as $BaSO_4$. If care is taken to have the solution cold and the volume about 300 cc. when neutralized with HCl, there is no danger of contamination of precipitate with silica. If such contamination occurs, the residue after ignition must be treated with a few drops of hydrofluoric acid and one drop of sulphuric acid. This is evaporated to dryness and the crucible re-ignited. A blank determination is run on the regeants employed.

$$\text{Factor } \frac{S}{BaSO_4} = 0.1374 \quad \text{Log. } \bar{1}.13793$$

DETERMINATION OF SILICA.

Procedure[1].

One gram of 100-mesh soil is mixed with 5 grams of sodium peroxide in an iron or nickel crucible. If the soil is low in organic matter .05 gram of starch is mixed with the soil before the peroxide is added. The mixture is heated carefully, the flame of the Bunsen being directed upon the charge and upon the side of the crucible until the action starts. The crucible is covered until the reaction is over and then kept at a dull red heat for 15 minutes. The contents of the crucible are transferred to a casserole and the crucible is washed out with a little hot water. The casserole is covered with a watch glass, and 75 cc. HCl (HCl, sp. gr. 1.2 diluted with 2 parts of water) are added through the lip. The mixture is evaporated on the water bath with occasional stirring until crumbling starts when 15 cc. HCl (1 part HCl, sp. gr. 1.2, to 1 part water) are added, the casserole is covered with a watch glass and digested on water bath for 10 to 15 minutes. Ten cc. of water are added and the silica is filtered with suction upon an 11-cm. ashless paper and washed with a hot solution of 5 cc. HCl, sp. gr. 1.2, to 95 cc. of water. The filtrate is evaporated to dryness and the residue dehydrated in the oven at 110° C. for 2 hours. The residue is taken up with 10 cc. of HCl (1 part HCl, sp. gr. 1.2, to 1 part water), covered and digested on water bath from 10 to 15 minutes. Forty cc. of water are added and the silica is filtered immediately onto 9-cm. ashless paper and washed with cold dilute HCl (1 cc. concentrated HCl to 99 cc. water). Paper and silica from second dehydration are placed in 35-cc. platinum crucible and the paper is carefully burned off. Paper and silica from the first dehydration are added and the silica is ignited to constant weight over Scimatco burner. Ten cc. of hydrofluoric acid and a few drops of sulphuric acid are added to crucible and the silica is volatilized by evaporation to dryness. The residue is ignited and weighed. Loss in weight represents SiO_2.

[1] Adapted from method of Lehner and Truog, J. Am. Chem. Soc., Vol. 38, pp. 1050-1063.

DETERMINATION OF CARBONATE CARBON.

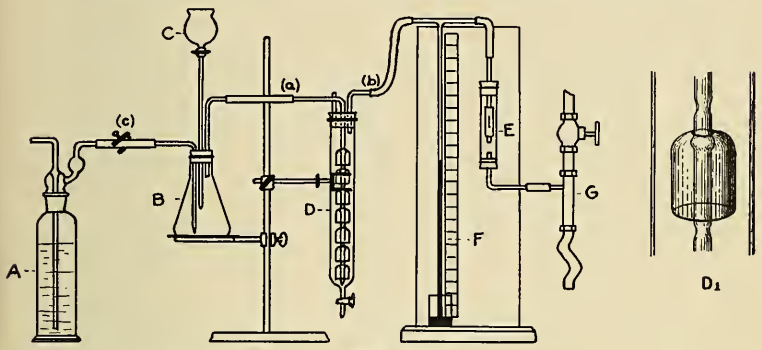

FIG. 2.

A—Gas Washing Bottle (Containing 30% KOH solution).
B—Wide Mouth Erlenmeyer Flask (200 cc. capacity). C—Extraction Funnel.
D—Absorption Tube, (Shown in detail at D_1). E—Bunsen Valve.
F—Mercury Suction Gauge. G—Suction Pump.

The method consists in the decomposition of the carbonates present in the sample by means of 1:10 HCl *in vacuo*. The CO_2 is expelled by means of a current of air and absorbed in standard NaOH solution. Barium chloride is added and the excess alkali titrated with standard HCl.

The absorption tube, which was designed and made in this laboratory, is very efficient and has an advantage over a tube containing glass beads in that it is more easily washed free from alkali.

Procedure.

Ten grams[1] of 100-mesh soil are placed in flask B and this connected up as shown in Fig. 2. Screw clamp (c) is closed. Absorption tube D is charged with 25 cc. of N/4 NaOH, measured by means of automatic overflow pipette, together with sufficient CO_2-free distilled water to cover the floats and a few drops of phenolphthalein indicator. Forty cc. of 1:10 HCl are run in from funnel C and the suction turned on very gradually until it equals 18 inches of mercury. Screw clamp (c) is then opened sufficiently to allow a slow current of air to pass through the apparatus (30 to 40 bubbles per minute in bottle A). The air current is continued with frequent agitation of flask for 30 minutes, when the suction is shut off and the absorption tube disconnected at points (a) and (b) and removed from the clamp. The contents of the

[1] On soils containing over 0.25% carbonate carbon a 5-gram sample is used.

absorption tube are run into a 300-cc. Erlenmeyer flask and the tube is washed three times with 40-cc. portions of CO_2-free water, run into tube through hole in stopper. During washing the tube is revolved sufficiently to wash the inner surfaces of the absorbing floats. Five cc. of neutral 10% $BaCl_2 \cdot 2H_2O$ solution are added and the solution titrated with N/10 HCl, using phenolphthalein indicator. A blank is run using the same amounts of reagents and water.

Percent of Carbonate Carbon = .012 (Titration of Blank — Titration of Sample.)

Duplicate should check within 0.1 cc. equivalent to .0012% on a 10-gram sample.

DETERMINATION OF LIME REQUIREMENT.

The method in use in this laboratory for the determination of the lime requirement is a combination of the method of Veitch[1] with that of Truog[2]. The principal objection to the Veitch determination as ordinarly carried out is the number of determinations that must be run before the correct end point is obtained. It has been found possible to eliminate one-half or more of these determinations by first running a Truog test on the sample and comparing the test paper obtained with a chart composed of test papers from a series of soils of varying lime requirements previously determined by the Veitch method.

Reagents.

Truog Test Solution.—200 grams of neutral calcium chloride are dissolved is water, 25 grams of neutral zinc sulphide (Merck's B. L. Reagent) added, and solution is diluted to 1 liter.

Lead Acetate Paper.—Filter paper is soaked in 10% lead acetate, dried in oven at 90° C. and cut into half-inch strips.

Standard Calcium Hydrate Solution.—N/50 $Ca(OH)_2$ solution prepared and preserved in bottle fitted with soda-lime guard tube. One cc. is equivalent to 200 pounds $CaCO_3$ requirement on 2,000,000 pounds of soil.

Phenolphthalein Indicator.—5% solution of phenolphthalein in 95% alcohol.

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[1] Jour. Am. Chem. Soc., Vol. 26, p. 661.

[2] Bulletin 249, Wisconsin Agr. Exp. Sta.

Procedure.

To 10 grams of 2-mm. soil in 300-cc. Erlenmeyer flask are added 5 cc. of Truog test solution and 95 cc. of water. Contents of flask are shaken and brought to boiling and boiled one minute, when a dry strip of lead acetate paper is laid on the mouth of flask and boiling continued for two minutes. The darkening produced on the paper is then compared with the test paper chart and the number of cubic centimeters of N/50 $Ca(OH)_2$ required to neutralize a 10-gram sample estimated. Five 10-gram samples of 2-mm. soil are weighed and treated in porcelain evaporating dishes with such amounts of N/50 $Ca(OH)_2$ that they form a series from 2 cc. below the estimated amount to 2 cc. above this amount, each member of the series varying by 1 cc. from the next. These are immediately evaporated to dryness, taken up with water and transferred to a 300-cc. flask. The volume should be about 150 cc. After shaking frequently for one hour, the flasks are allowed to stand over night and 50 cc. of the supernatant liquid of each is transferred to a small beaker. Phenolphthalein is added and the contents of the beaker are boiled to one-third their original volume. By noting which solutions in the series turn pink, the lime requirement is estimated to within 100 pounds of calcium carbonate. One cc. N/50 $Ca(OH)_2$ equals 200 pounds calcium carbonate requirement on 2,000,000 pounds of soil.

DETERMINATION OF NITRATE NITROGEN.

Reagents.

Phenol-di-sulphonic Acid.—75 grams of pure crystallized phenol are mixed with 920 grams (500 cc.) of concentrated H_2SO_4 (sp. gr. 1.84) and heated for six hours at 100° C. by placing the lightly stoppered flask in boiling water. Acid so prepared is stored in brown glass bottle.

Standard Color Solution.—A solution containing 0.1 mg. N per cc. is prepared by dissolving 0.7215 gram dried c. p. KNO_3 in water and diluting to one liter. One hundred cc. of this solution are removed and diluted to 1 liter. This solution contains .01 mg. N as nitrate per cc. The color standard is prepared by evaporating 10 cc. of the solution, treating with phenol-di-sulphonic acid and proceeding as in the following method.

Procedure.

One hundred grams of 2-mm. soil, together with 1 gr $CuSO_4$ and exactly 250 cc. distilled nitrate-free water placed in dry soil-shaking bottle. This is stoppered and sh en in shaking machine for 30 minutes. Bottle is removed allowed to stand over night, when 75 cc. of the clear sup natant liquid are pipetted into a dry flask. Five-tenths gr of powdered MgO is added and the mixture heated to 60° with occasional shaking. The flask is closed with a rubb stopper and allowed to cool with further shaking. The sol tion is filtered through a dry filter into a dry flask, the f few cc. of filtrate being discarded. Ten cc. of clear filtr are evaporated on the water bath in porcelain dish. The re due is treated with 2 cc. of phenol-di-sulphonic acid an mixed thoroughly by scratching with a glass rod. After 1 minutes 15 cc. of water are added, followed by enoug NH_4OH (1:1) to produce a yellow color, and the solution transferred to a 250 cc. flask and made up to the mark. T is shaken and, if not perfectly clear, the solution is filte The solution is compared in a Dubosc colorimeter with standard color solution and the result calculated as follov

Mg. of N as nitrate per 100 grams soil = 0.05 × read on scale of standard solution (unknown being se 50 on scale or calculated to that amount).

DETERMINATION OF AMMONIUM NITROGEN[1].

Solutions.

Standard acid and alkali solutions required as for nitrogen.

Procedure.

One hundred grams of 2-mm. soil are placed toge with 5 grams of magnesium oxide, 300 cc. of water, and a drops of hydrocarbon oil in a liter copper distillation fl The mixture is distilled into 10 cc. of 5/14 normal H_2SO_4 in the distillate measures 250 cc. The distillate is boiled a titrated after cooling with N/14 NaOH, using alizarin indicator. One cc. N/14 NaOH = 1 milligram of nitrogen.

[1] This method is not recommended for absolute amounts of ammonia but has been found very satisfactory for determining relative amounts.